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This grant was sponsored by the FY95 Defense University Research Instrumentation Program (DURIP). The objective was to acquire instrumentation compatible with the Duke University Free Electron Laser (FEL) Laboratory light sources in the infrared (IR), ultra-violet (UV), extreme ultra-violet (XUV), X-Ray and gamma-ray wavelength regions, that would support the research and characterization of advanced materials, including nano-modulated coatings and ceramics. This report summarizes progress made with the existing FEL Laboratory light sources and describes three major pieces of equipment fabricated with funds from this program for application to nano-modulated coatings and ceramics research. Capabilities of the resulting equipment and experiments planned in the near-term are also described.

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FELL-DURIP95-313-6015-98

Instrumentation for Processing and Characterization of Nano-Modulated Ceramics

Final Technical Report

AFOSR Grant No. F49620-95-1-0476

Principal Investigator:

Professor Vladimir N. Litvinenko

March 12, 1998

Period of Performance:

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Instrumentation for Processing and Characterization of Nano-Modulated Ceramics

Vladimir N. Litvinenko, FEL Laboratory, Duke University

ABSTRACT

This grant was sponsored by the FY95 Defense University Research Instrumentation Program (DURIP). The objective was to acquire instrumentation compatible with the Duke University Free Electron Laser (FEL) Laboratory light sources in the infrared (IR), ultra-violet (UV), extreme ultra-violet (XUV), X-Ray and gamma-ray wavelength regions, that would support the research and characterization of advanced materials, including nano-modulated coatings and ceramics. This report summarizes progress made with the existing FEL Laboratory light sources and describes three major pieces of equipment fabricated with funds from this program for application to nano-modulated coatings and ceramics research. Capabilities of the resulting equipment and experiments planned in the near-term are also described.

BACKGROUND

This report documents the results of a grant received by Duke University from the FY95 Defense University Research Instrumentation Program (DURIP) for the acquisition of *Instrumentation for Processing and Characterization of Nano-Modulated Ceramics*. This grant, amounting to $299,000, was administered by the Air Force Office of Scientific Research (AFOSR); Dr. Alexander Pechenik served as AFOSR's Program Manager.

DEPARTMENT OF DEFENSE AREAS OF INTEREST

The instrumentation proposed for this grant was aimed at establishment of new research capabilities to the following Department of Defense (DoD) areas of interest (Section VII of the BAA 95-2):

- Processing and characterization of the nano-modulated oxide coatings with the scale of compositional layers ranging from a few nanometers to a few microns;

- Characterization of oxide coating hardness, toughness and resistance to wear and corrosion;
- Characterization of HfO_2 coatings as a promising candidate for protection of aircraft canopies and seeker windows; and
- Time-resolved in-situ characterization of optical properties of evolving nano-scale metal and oxide structures.

DUKE STORAGE RING LIGHT SOURCE CAPABILITIES

The capabilities for supporting the proposed research have improved substantially since submission of the proposal for this program and in parallel with the execution of this grant. These improvements were funded mostly by other DoD programs (U. S. Office of Naval Research Grant No. N00014-94-1-0818, "A Center for Fundamental Surgical, Structural, Photochemical, and Spectroscopic Studies using IR, UV, and X-ray Free Electron Lasers"; and Air Force Office of Scientific Research Grant No. F49620-93-1-0590, "Research on the Physics of Ultra-High Brightness, Ultra-Relativistic Electron Beams").

The Duke storage ring operates in the 0.25 GeV - 1.1 GeV energy range with maximum stored current of 150 mA [1] . Installation and commissioning of the OK-4 FEL system was completed in November 1995. The OK-4 FEL can produce tunable spontaneous radiation from 120 nm to 600 nm. Since November 1996, the OK-4 FEL has produced high power coherent radiation in UV range from 345 to 413 nm, as well as beams of gamma-rays with tunable energy from 3 MeV to 16 MeV [2, 3]. In very near future, the OK-4 FEL will be configured to produce laser energy in deep UV range (down to 190 nm). These capabilities are sufficient for initial characterization of optical properties of nano-coatings under study.

PROGRESS

Instrumentation was acquired and fabricated for application of the existing and evolving Duke University Free Electron Laser Laboratory (FEL) storage ring light sources to studies of nano-structures and nano-ceramics. The list of equipment included: a vacuum chamber and deposition system; in-situ interferometry, holography and reflectometry equipment; and real-time data acquisition equipment.

Most of proposed equipment has been assembled and tested. The remainder of the standard equipment has been ordered and will be assembled in April, 1998. Full capabilities of the system will be established by the Summer of 1998 and first experiments with nano-structures will start by that time.

The following sections summarize the progress made with acquisition, assembly and test of the equipment.

VACUUM CHAMBER AND DEPOSITION SYSTEM

The vacuum chamber and deposition system includes a multi-port ultra-high vacuum chamber/deposition assembly, an ultra-high vacuum pump-cart, and interface assemblies as needed to support specific experiments.

The multi-port ultra-high vacuum assembly provides the necessary elements for processing and characterizing nano-modulated coatings. A photograph is shown in Fig.1. This assembly is equipped with five optical ports, a port for the deposition system, a port for the ultra-high vacuum pump, and five ports for vacuum pumping, gas controllers and vacuum gauges. Currently, CaF_2 windows are installed for optical transparency down to VUV (120 nm). The

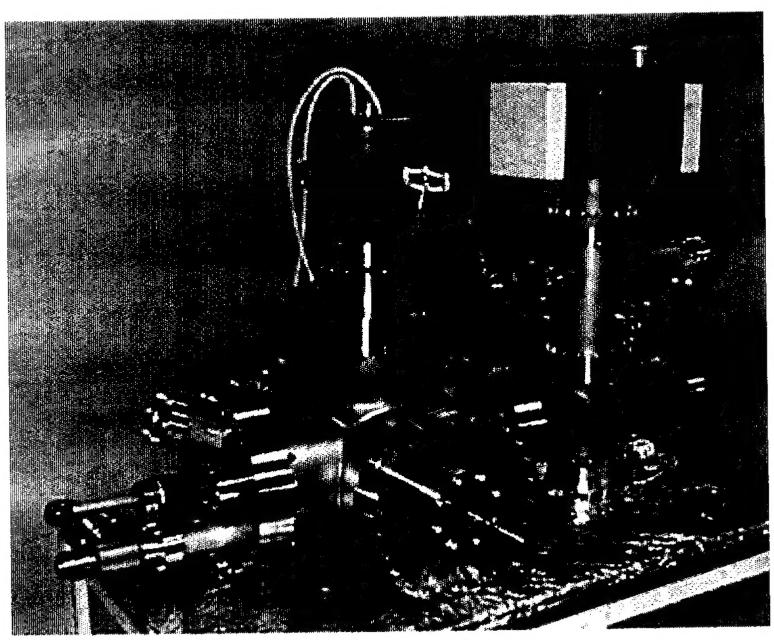

Figure 1. Photograph of the multi-port ultra-high vacuum chamber/deposition assembly for processing and characterization of nano-modulated coatings.

apparatus includes screens to protect windows and other essential parts of the equipment during the process of coating. The assembly has been built and is currently undergoing tests; so far, it has demonstrated ability to hold a very high vacuum (10^{-10} torr range).

The ultra-high vacuum pump-cart has been completed and provides in-situ pumping of the vacuum chamber/deposition assembly. The pump-cart, partially funded by this grant, is shown in Fig.2. The cart incorporates an oil-free turbo-pump, a dry piston pump, and a residual gas analyzer. The residual gas analyzer, together with tanks for gasses such as nitrogen, oxygen and argon, will control and monitor the atmospheric environment during both coating deposition and characterization tests.

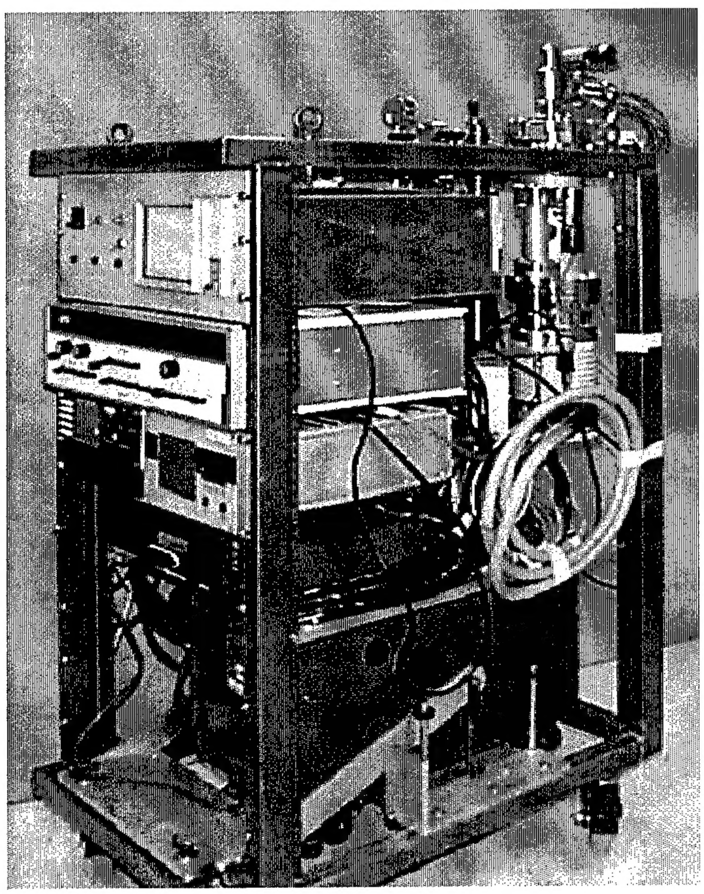

Figure.2. Photograph of the oil-free ultra-high vacuum pump-cart for in-situ control of the multi-port vacuum chamber/deposition assembly.

Finally, a small portion of funds from this grant has provided an ultra-high vacuum interface for connecting the above equipment to the storage ring light sources.

IN-SITU INTERFEROMETRY, HOLOGRAPHY AND REFLECTROMETRY EQUIPMENT

The equipment for the laser interferometry and reflectometry includes a set of conventional lasers and a set of time-resolved, phase sensitive electronics. In addition to conventional lasers, we have developed a new method utilizing spontaneous radiation from the OK-4 for real-time reflectometry. The equipment purchased for laser reflectometry (lock-in amplifiers, detection system, net-work analyzer) has been utilized for this novel method. This method of reflectivity measurement uses tunable OK-4 radiation and, therefore, provides for full coverage of the spectral range. A conventional nitrogen laser purchased on this grant and existing helium-neon lasers will be used for holography, standard tests of the samples, and for alignment of the optical elements for nano-ceramics experiments.

REAL-TIME DATA ACQUISITION EQUIPMENT

The real time data acquisition system is fully computer controlled and incorporated into the Duke storage ring. It is comprised of two computers connected via Ethernet with the main control room, a 4-Ghz LeCroy oscilloscope , two CAMAC crates with real time data acquisition hardware, and controllers located in two racks. All real time data acquisition system is connected by comprehensive cable net-work and is fully operational. The control system of the nano-ceramics experiments includes a set of computer controlled stepper-motor drivers. The piezo drivers are utilized for components requiring high precision of alignment.

At the present time, in-situ detection for XUV radiation utilizes two monochrometers (funded by other grants) and a number of time-resolved detectors. Additionally, a UV streak-camera (funded by other grants) will be available after April, 1998.

INTEGRATED NANO-CERAMICS RESEARCH SYSTEM

The full nano-ceramics research system will be located at the optical table in South-East optical room of the Duke FEL Laboratory (see Fig.3). A set of two optical tables, three bread-boards and set of supporting equipment (tables, holders, mirrors, lenses, diaphragms, etc.) are used to support the equipment for characterization of nano-ceramics. All the equipment for reflectometry and transparency measurements is in place and operational. The temperature

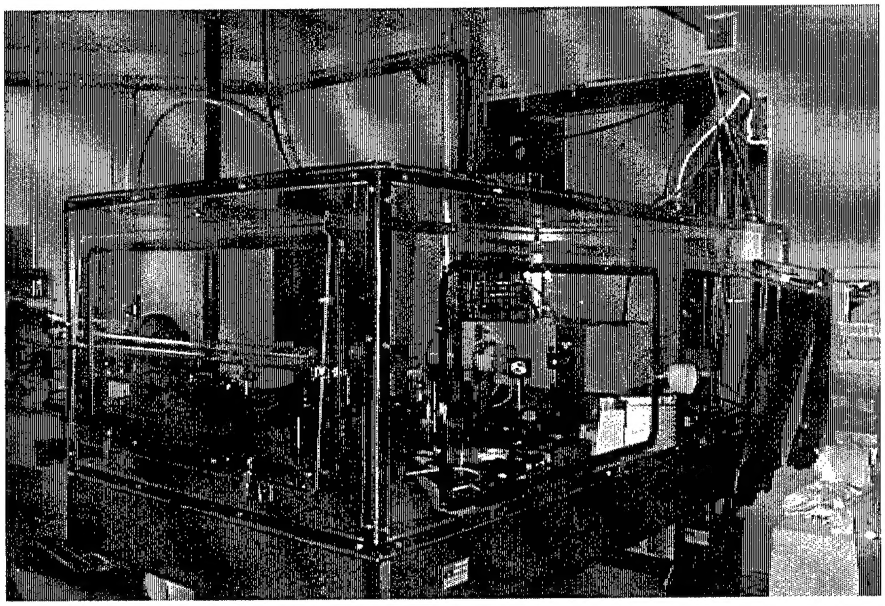

Figure 3. The view for the South-East optical room where the integrated nano-ceramics research system is being situated.

sensors have been delivered and tested. The interferometer will be assembled in situ when the vacuum apparatus passes all tests. The differential pumping section has been designed, purchased and is in the process of assembly.

The OK-4 FEL spontaneous and coherent radiation as well as gamma-ray beams are delivered to this room by an ultra-high vacuum beam-line. The OK-4 FEL diagnostics and the real-time data acquisition system are located in this room. In addition, the photo-emission electron microscope (PEEM) with nm-range resolution (supported by the ONR Grant) is located in the same room. It provides for very unique and additional capabilities for studies of the nano-structures.

PROGRESS IN PROCESSING AND CHARACTERIZATION OF NANO-MODULATED OXIDE COATINGS

We have a number of substrates in hand for testing nano-modulated coatings. In the absence of the multi-port ultra-high vacuum system during 1996-1997, we used existing vacuum system of the OK-4 FEL to study multi-layer nano-scale coatings. Our studies confirmed our expectations that in vacuum conditions and in the presence of high intensity XUV radiation, a

layer of HfO_2 coating protects nano-coatings from optical damage. We plan to confirm this result in the presence of controlled atmospheres using the new apparatus.

CONCLUSION

The most essential parts of the Instrumentation for Processing and Characterization of Nano-Modulated Ceramics have been built and tested. A few remaining pieces of standard electronic and vacuum equipment will be delivered to Duke FEL laboratory very shortly. The apparatus required for the proposed research will be ready for the use by Summer of 1998. Thanks to an extension of the Grant period, we were able to use the available budget for most important and essential elements of the system, avoiding duplication with equipment supported by other grants and contracts. As the result, we have system capabilities that reach far beyond our expectations.

In combination with the rest of the OK-4 FEL equipment and its user station, the new system will allow the study of new nano-structures and nano-ceramics such as wide-gap semiconductors and quantum dots. It is apparent that the presence of the PEEM apparatus next to the new nano-ceramic apparatus provides us with capabilities to study electronic phenomena of sub-nano-scale layers.

In near future we will start to use new apparatus for test of stability of aluminum nano-layers in the ultra-high vacuum environment and high intensity of XUV radiation. We will also continue to study of the radiation stability of multi-layer nano-scale oxide coatings with and without HfO_2 protective nano-layer. We will use new apparatus for these studies.

In combination with available high power OK-4 UV radiation and desirable mixture of gases (oxygen, nitrogen, CO, etc.) the new apparatus will imitate the extreme conditions for aircraft canopies and seeker windows at high altitudes. This experiment can be initiated during 1998, provided that samples of materials used for aircraft canopies and seeker windows and information of the gas mixture typical for high altitudes can be made available to the Duke FEL Laboratory.

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